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N87-16773 198
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1986

NASA/ASEE FACULTY FELLOWSHIP PROGRAM

MARSHALL SPACE FLIGHT CENTER

EVALUATION OF MOISTURE BARRIER COATINGS
ON CARBON-PHENOLIC SRM
NOZZLE MATERIALS

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Date:	August 22, 1986
Contract No.:	NGT 01-002-099 The University of Alabama

XXXI

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ABSTRACT

The carbon-phenolic composite ablative material used on the Solid Rocket Motor Nozzle is known to absorb moisture from the atmosphere. This could cause problems such as pocketing during firing. Part of this work was to test several moisture barrier coatings on the SRM nozzle material. This report shows data on six of about twelve coatings to be tested. The data were obtained from immersion of coated samples in an environmental chamber at 100°F and 100% relative humidity and by using a modified TGA (thermal gravimetric analysis) technique. The TGA technique involved allowing wet nitrogen (25°C, 80% relative humidity) to flow across a small sample at about 65 cm³ per minute while continually monitoring the weight increase. These preliminary results show Kel-F-800, a material supplied by 3M Corporation to be the better moisture barrier.

A second task was to collect data on the relative absorption of water and kerosene into the carbon-phenolic SRM nozzle material. These data indicate that water absorbs into the nozzle material to a much greater extent than kerosene. Thus kerosene is the more likely solvent in which to make specific gravity measurements on the SRM nozzle material.

ACKNOWLEDGEMENTS

I want to thank Mr. R. L. Nichols and Dr. R. G. Clinton for offering me a second summer appointment. I want to express my appreciation to my NASA co-worker, Mrs. Linda Jeter, for beginning this work, for working with me, and for continuing the project after my ten weeks are completed. Everyone in the Non-Metallics Division has been extremely helpful. I must, however, mention some people who have gone out of their way to help: Mr. Donald Morris, without whom very little could have been accomplished; Mr. Roger Harwell and Mr. David Webb, who instructed us in coating techniques, among other things; Mr. Benjamin Goldberg who willingly shared his ideas; and Mr. Robert Durham for a marvelous suggestion on how to coat small samples.

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INTRODUCTION

The ablative material used on the solid rocket motor nozzle is a carbon-phenolic composite known to absorb water from the atmosphere, especially under warm humid conditions. This absorbed water could cause problems such as pocketing during firing or it could cause general degradation of the composite over a period of time. (References)

This work should test the efficiency of various coating materials which could be used as moisture barriers on the surface of the carbon-phenolic composite. The carbon-phenolic materials from two 404 rings were obtained from Morton-Thiokol Company. Samples were cut out and machined into two sizes 1" x 1" x 2" for exposure to 100% relative humidity at 100°F in a controlled humidity chamber and 1/8" cubes for a modified TBA (Thermal Gravimetric Analysis) analysis. This modified TBA analysis consisted of allowing wet nitrogen gas flow isothermally across the sample for about two days while monitoring the weight gain.

A secondary task was to immerse carbon-phenolic samples into water and in kerosene to compare the relative absorption of each into the carbon-phenolic material.

OBJECTIVES

The objectives of this work were to:

1. Subject coating materials from several candidates to conditions of high humidity, and possibly recommend one or more as moisture barriers with which to coat the carbon-phenolic SRM nozzle material.
2. Obtain data on the relative absorption of water and kerosene into the carbon-phenolic SRM nozzle material.

PROCEDURES

The material to be tested was obtained from Morton-Thiokol Company. The gross samples (carbon-phenolic nozzle material) were from two 404 rings of the SRM. They consisted of eight blocks, four from each ring. Each of the four blocks represents about 45° taken from alternate 45° sections around the ring (Fig. 1). Blocks A, B, C, & D came from ring number 42. Blocks E, F, G, & H came from ring number 45. Samples were taken in such manner as to get representative material from each block. The samples to be tested at 100°F, 100% relative humidity were cut to 1" x 1" x 2", a convenient size for possible diffusion studies and consistent with the amount of material available, coating case, etc. The small samples (1/8" cubes) were cut to be compatible with the TGA apparatus. The 1/8" cubes were also used to test relative absorption of water and kerosene into the carbon-phenolic material. It should be noted that this material was not the same as that used in the TGA and humidity chamber work but was from nozzle material already on hand.

Mixing, curing and coating procedures followed were generally those set down by the suppliers. Duration of exposure in the humidity chamber has not been determined because data acquisition will continue for at least another month beyond the date of this report. The time limit on TGA analysis was determined by what was reasonable in terms of available instrument time and the fact that total immersion and TGA data were very similar for about two days (Fig. 2). The only technique, other than essentially standard procedures, was coating the small cubes. They were lightly adhered to two-sided tape on an aluminum plate. Five exposed sides of the cubes were coated and cured. The samples were turned to expose the uncoated side which was then coated, cured and the samples placed in a dry environment to await testing.

The procedure followed in weighing the small cubes immersed in water and in kerosene was to remove each individually with tweezers, touch the sample to absorbent tissue quickly to remove adsorbed solvent, weigh, and return it to the solvent container. Most of these tests were done using three replicate samples.

RESULTS AND DISCUSSION

There were originally eleven moisture barrier candidates (Table 1 and 2). The time available allowed data to be reported on six of these. A complete report on all coatings listed (possibly more) will probably be available in November 1986. Table 3 shows data on the absorption of moisture by the coatings themselves; only one, scuffcoat, absorbed significant amounts of water. Poly/Ep and Kel-F-800 absorbed moderate amounts but other data (Fig. 3 and 4) showed them to be fairly good moisture barriers, especially Kel-F-800. Figure 3 compares the weight gain for each of the six coatings tested to that for an uncoated sample using the TGA apparatus. All coatings absorbed less moisture than the uncoated control. Three coatings, Poly/Ep, Desoto Green, and Kel-F-800 appeared to be about equally efficient moisture barriers in this test. However, Kel-F-800 is the best moisture barrier considering the results from the environmental chamber (100°F, 100% Rel. Hum.) shown in Figure 4. An interesting feature of the results shown in Figure 4 is the fact that the uncoated control absorbs moisture at a slower rate after a couple of days than two of the coated samples. One explanation could be that the uncoated control more nearly follows Fick's law of diffusion than the coated samples.

Figure 5 concerning the relative absorption of kerosene and water into carbon-phenolic material shows water to absorb to considerable more than kerosene. One of the concerns here was which solvent would be preferable for specific gravity measurements. These data (Fig. 5) indicates kerosene to be the solvent of choice. Further tests will be done weighing the samples in the two solvents over a period of time using a modified Westphal balance.

CONCLUSIONS AND RECOMMENDATIONS

Considering the data available, Kel-F-800 is the best moisture barrier of the six tested so far. The coating material is a copolymer of chlorotrifluoroethylene (CTFE) and vinylidene fluoride (VF_2) supplied by the 3M Corporation. This coating has a long pot-life and is easy to apply.

Considering water and kerosene as solvents in which to do specific gravity measurements on carbon-phenolic SRM nozzle material the data shows kerosene to be the more likely choice. One point brought out in oral presentation was the need to perform flame tests on the coating materials. It is recommended that this be done before a final selection is made.

TABLE 1

LIST OF COATINGS TO BE TESTED

1. Poly/EP Polyamide Epoxy Coating
(Degraco Company)
2. EPON RESIN 828/TETA
(Shell/Union Carbide)
3. Fluorad Brand Surface Modifier FC-723
(3M Corporation)
4. KEL-F Brand 800 Resin
(3M Corporation)
5. Fluorad Brand Conformal Coating FC-725
(3M Corporation)
6. Integral Fuel Tank Coating, Green
(DeSoto, Inc.)
7. Fuel Barrier Coating 473-13
(Sikkens)
8. Hysol Adhesive EA 934 Non-Asbestos
(Dexter Hysol Aerospace and Industrial Adhesives)
9. Chemglaze Z 302
(Lord Chemical Products)
10. Scuffcoat
(Mixture of Products)
11. EXXON Butyl Rubber

TABLE 2

LIST OF COATING MATERIALS

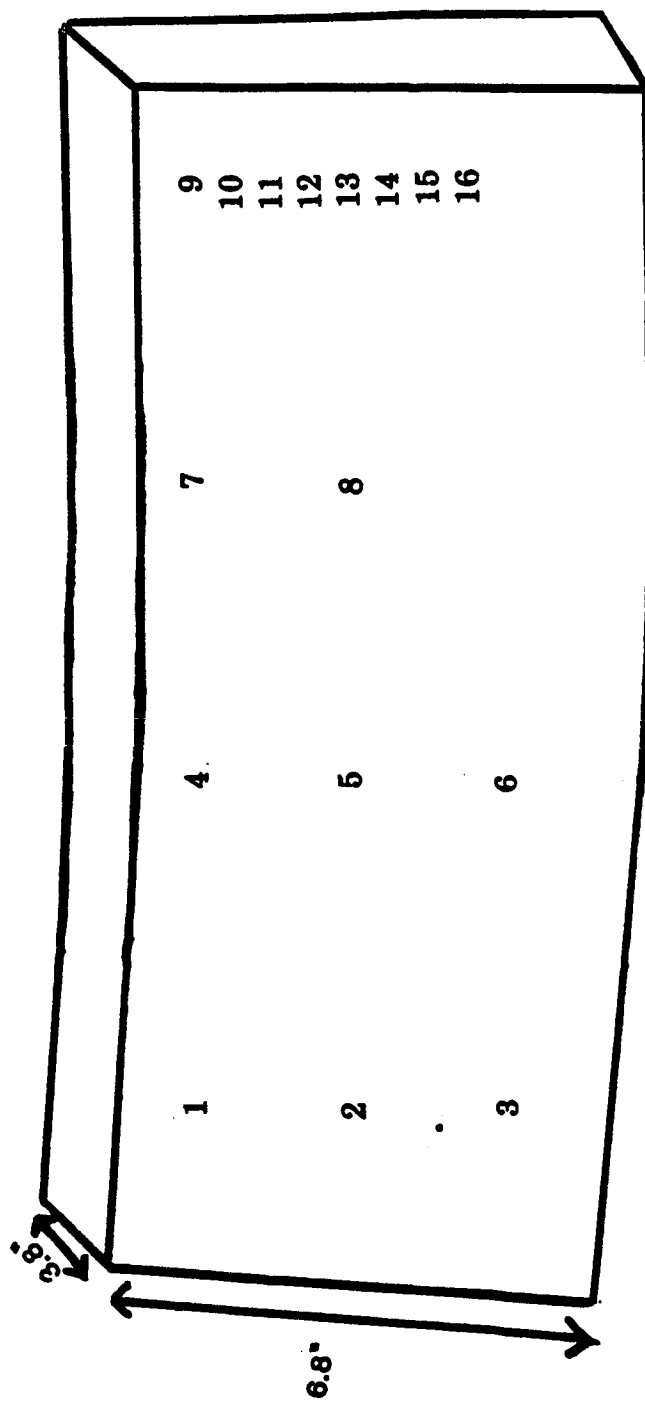
1. Poly/EP Polyamide/Epoxy thermosetting coating.
2. Epon Resin 828/TETA
Epon 828 a reactive light colored liquid epoxy resin.
TETA Tetraethylene-tetramine.
3. FC-723 is an oleophobic-hydrophobic fluorochemical polymer dissolved in a fluorinated inert material.
4. KEL-F 800 is a copolymer of chlorotrifluoroethylene (CTFE) and vinylidene fluoride (VF₂).
5. FC-725 is a clear mobile solution of a fluorine containing polymer that dries to a transparent acrylic coating.
6. Integral Fuel Tank Coating Green, is a chemically cured acid resistant and fluid resistant urethane.
7. 473-13 is a two component high build epoxy system.
8. EA 934 NA consists of a gray thixotropic paste and an amber liquid amine curing agent.
9. Chemglaze Z302 is an oil-free, moisture curing polyurethane.
10. Scuffcoat is a blend. The base is Epon 828 with NER-010 and the curing agent is Versamid 115 and 125. Epon 828 is a reactive light colored liquid epoxy resin. NER-010 is a mixture of glycerol di- and tri- glycidyl ether. Versamid 115 and 125 are polyamide resins.
11. EXXON Butyl Rubber is a complex material containing EXXON BUTYL 268, zinc oxide, stearic acid, N-770 Black, Hydrogenated Resin Ester, Sulfur, P-Quinone dioxime, Lead oxide, Heptane (solvent), and Isopropanol.

TABLE 3

WEIGHT GAIN FOR COATINGS ON ALUMINUM PLATES

4" x 4" Coated Al Plates
Placed in Chamber 100°F 100% Relative Humidity

Coating	Percent Wt. Gain (Based on Wt. of Coating)	Days in Chamber
Poly/EP	4.61	30
DeSoto Green	0.34	28
473-13	0.87	28
KEL-F-800	4.22	23
Scuffcoat	10.40	21
Chemglaze-Z302	1.07	18



SAMPLES 1-8, DIMENSIONS 1" X 1" X 2"

SAMPLES 9-16, DIMENSIONS 1/8" CUBES

FIGURE-1. TYPICAL SECTION OF BLOCK OF CARBON-PHENOLIC MATERIAL FROM WHICH
SAMPLES TO BE COATED WERE TAKEN AND MACHINED.

Sample: CP/NO COATING/TR6

Size: 65.54 mg

Rate: 00 DEG/MIN/N2

Program: TGA Analysis V2.0

TGA

Date: 11-Jul-86 Time: 13:22:34

File: CP.06 TGA RCM 1

Operator: RCM

Plotted: 17-Aug-86 10:53:49

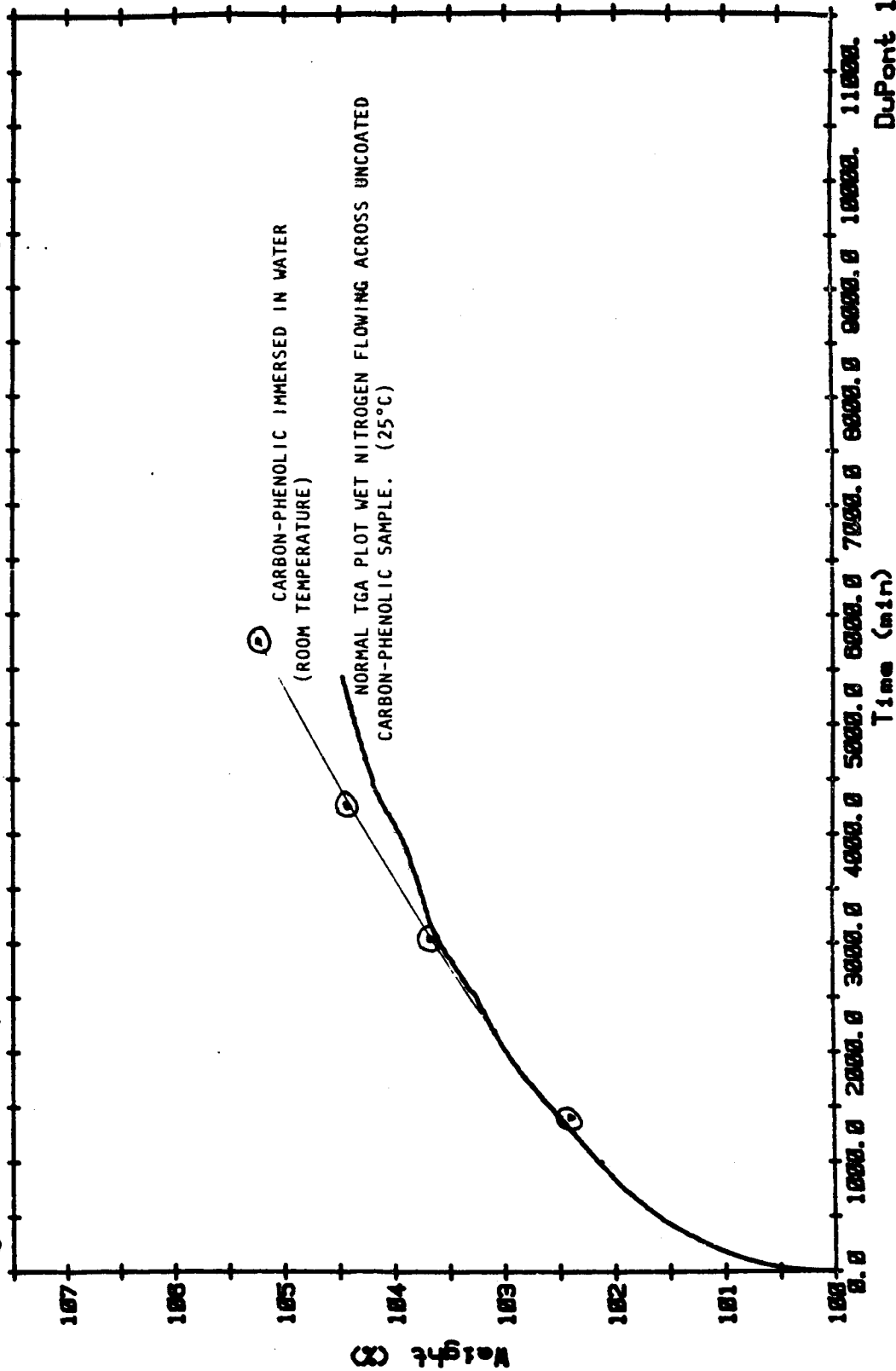


FIGURE 2. COMPARISON OF WEIGHT GAIN FOR CARBON-PHENOLIC MATERIAL IMMERSSED IN WATER (ROOM TEMPERATURE) AND EXPOSED TO WET NITROGEN (25°F, 80% REL. HUM., 65 CM³/MIN. FLOW)

TGA

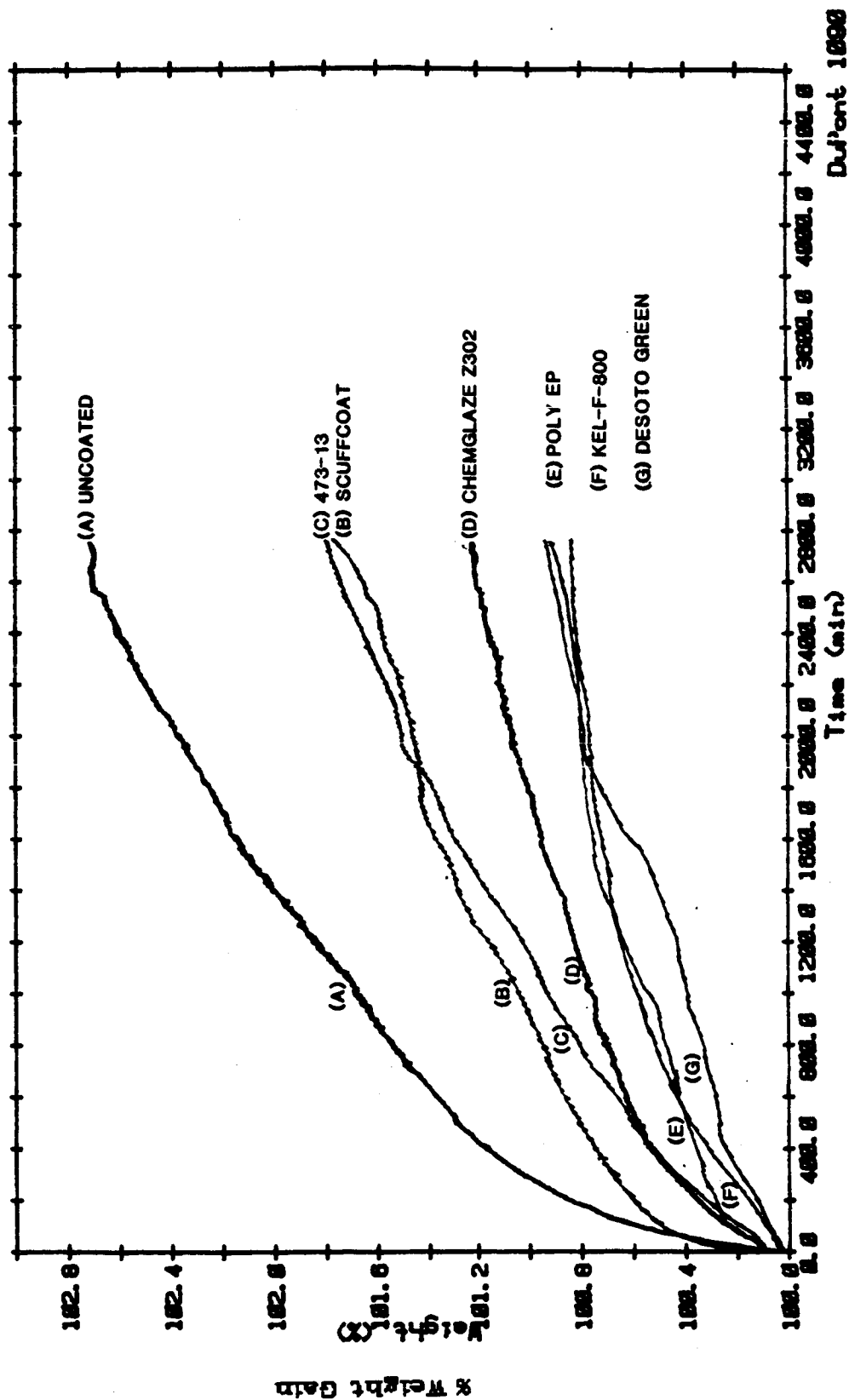


FIGURE 3. WEIGHT GAIN VS TIME FOR CARBON-PHENOLIC MATERIAL EXPOSED TO WET NITROGEN
(65CM³, 25°C, 80% REL. HUM.)

PERCENT WEIGHT GAIN VS TIME

Moisture Barrier Candidates

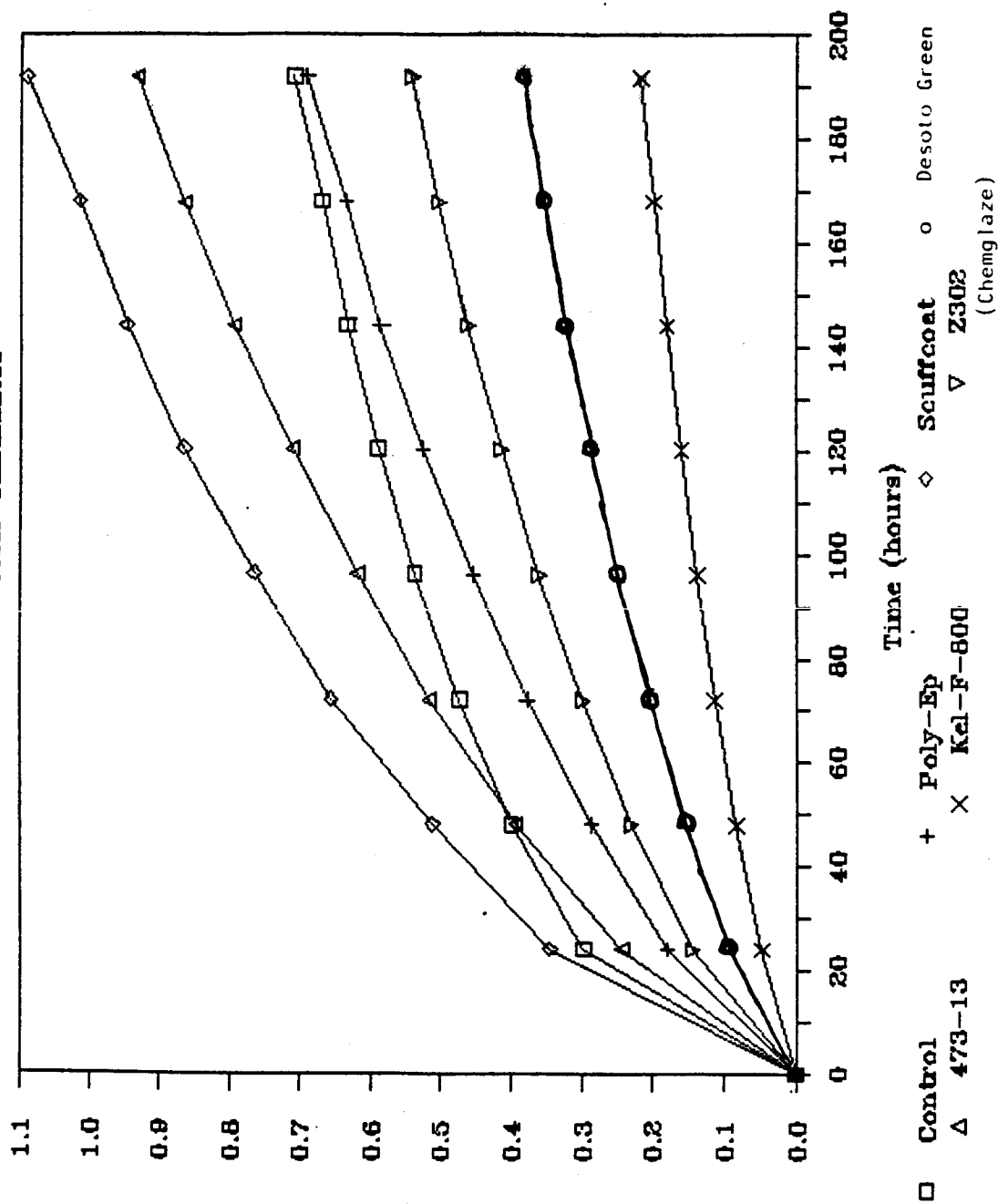


FIGURE 4. 1" x 1" x 2" CARBON-PHENOLIC BLOCKS IN ENVIRONMENTAL CHAMBER
(100°F, 100 REL. HUM.)

PERCENT WEIGHT GAIN VS TIME

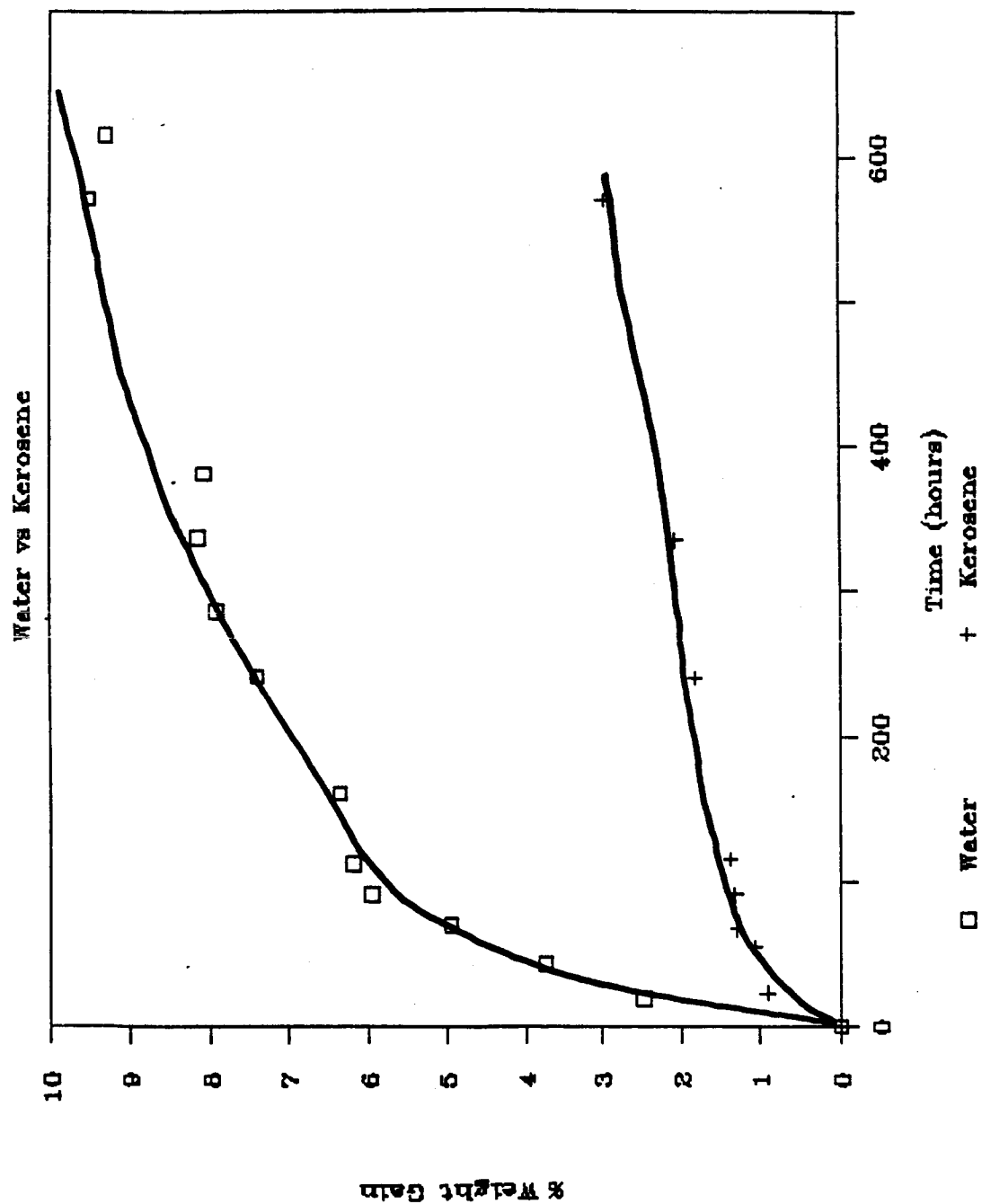


FIGURE 5. CARBON-PHENOLIC MATERIAL IMMERSIED IN KEROSENE AND IN WATER (ROOM TEMPERATURE.)

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